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European Patent Office
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(11) Publication number:

**0 061 894
A2**

(12)

EUROPEAN PATENT APPLICATION

(21) Application number: 82301556.5

(51) Int. Cl.²: C 10 L 1/14

(22) Date of filing: 24.03.82

(30) Priority: 31.03.81 GB 8110082

(54) Date of publication of application:
06.10.82 Bulletin 82/40

(84) Designated Contracting States:
AT BE CH DE FR GB IT LI LU NL SE

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(54) Two-component flow improver additive for middle distillate fuel oils:

(57) Distillate fuels, particularly those having a relatively high final boiling point, are significantly improved in their flow and filterability properties utilising a two component additive consisting of 25 to 95 wt.% preferably 50 to 90 wt.% of C₃₀-C₂₀₀ oil-soluble nitrogen compound being an amide or amine salt of an aromatic or cycloaliphatic carboxylic acid and 75 to 5 wt.% preferably 10 to 50 wt.% of a certain category of ethylene-vinyl acetate copolymers.

EP 0 061 894 A2

0061894

-2-

TWO-COMPONENT FLOW IMPROVER ADDITIVE
FOR MIDDLE DISTILLATE FUEL OILS

1 This invention relates to the use of certain mixtures of
additives to improve the flow and filterability properties
of distillate fuels at low temperatures to fuels containing
the mixtures and to concentrates of the additives for
5 incorporation into the fuel.

Particularly, the invention relates to an additive system
composed of a nitrogen-containing wax crystal growth inhibitor
and a particular category of ethylene-vinyl acetate copolymer.

Various additives are disclosed in the art for improving the
10 flow properties of middle distillate fuel oils. Combinations
of additives which function both as wax nucleators and/or
wax crystal growth stimulators and as wax growth arrestors
are well-known and disclosed for example in U.S. Patent
3,961,916 issued June 8th 1976 to Ilnycky et al, which
15 shows an additive combination comprising ethylene copolymerised
with ethylenically unsaturated mono- or dicarboxylic acid
alkyl esters or a vinyl ester of a C₁-C₁₇ saturated fatty
acid.

Additive systems comprising nitrogen containing amide or
20 amine salts as used in the present invention are disclosed
in U.S. Patent 4,211,534 issued July 8th, 1980 to Feldman

0061894

-3-

- 1 which discloses a three component combination additive
flow improver consisting of an ethylene polymer or
copolymer, a second polymer of an oil soluble ester
and/or C₃ and higher olefin polymer and, as a third
5 component, a nitrogen containing compound. This
three component system is said to have advantages over
combinations consisting of any two of the additive
components for improving the cold flow properties
of distillate fuels.
- 10 U.S. Patent 3,982,909, issued September 28th, 1976 to
Bollyday discloses an additive system comprising amides,
diamides and ammonium salts alone or in combination with
certain hydrocarbons such as microcrystalline waxes or
petrolatums and/or an ethylene backbone polymeric pour
15 depressant, the combination being useful as a flow improver
for middle distillate fuels.

Nitrogen containing oil soluble succinic acid or its
derivatives are disclosed in U.S. Patent 4,147,520
issued April 3, 1975 to Ilynckyj which describes these
20 materials in combination with ethylene vinyl acetate
copolymer wax nucleators.

The present invention is based on the discovery that a
two component additive system consisting essentially of an
amine salt that is an alkyl ammonium or amide compound
25 having a total of 30-200 preferably 50-150 carbon
atoms derived from certain carboxylic acids or

0061894

-4-

- 1 vinyl acetate copolymer is highly effective at relatively lower treatment levels for improving the flow and filterability properties of middle distillate fuels below their cloud points.
- 5 In accordance with the present invention there have been discovered improved wax containing petroleum fuel oil compositions comprising a wax containing middle distillate fuel oil, boiling in the range of about 120°C-500°C, which has been improved in its low temperature properties by the
- 10 addition of 0.005 to 0.5 wt.%, preferably 0.005 to 0.25 wt.% of a flow and filterability improver consisting essentially of:
- (a) In the range of about 25 to 95 wt.% preferably 50 to 90 wt.% based on a total weight of flow
- 15 improver of a C₃₀-C₃₀₀ oil-soluble nitrogen compound wax crystal growth inhibitor having at least one straight C₈-C₄₀ alkyl chain and being selected from the class consisting of alkyl ammonium salts and/or amides of aromatic or cycloaliphatic
- 20 polycarboxylic acids or anhydrides thereof or the amides/amine salts of partial esters, e.g. monoesters of said polycarboxylic acids, e.g. dicarboxylic acids, and

0061894

-5-

1 (b) In the range of 75-5 wt.% preferably 50-10 wt.%
of an ethylene-vinyl acetate copolymer having a
vinyl acetate content of about 10-40 wt.% preferably
10-35 wt.% and a number average molecular weight
5 (M_n) of about 1000-30,000 e.g. 1500 to 7000
preferably 1500 to 5500 and a degree of branching
in the range of about 1 to 20 preferably 2-12 methyl
groups per 100 methylene groups other than the
acetate groups as determined by Nuclear Magnetic
10 Resonance (1H NMR) Spectroscopy.

The flow improver combination of the present invention
is useful in a broad category of distillate fuels
boiling in the range of about 120°C to about 500°C
(ASTM D1160), preferably those distillate fuels
15 boiling in the range of about 150°C-400°C. The
invention is especially applicable to fuels which
have a relatively high final boiling point (FBP),
that is, above 360°C.

The use of such fuels has recently become more
20 extensive and these fuels tend to contain longer
chain n-paraffins and will generally have higher
cloud points. Generally speaking, these fuels are
more difficult to treat effectively with conventional
flow improver additives. The most common petroleum
25 distillate fuels are kerosene, jet fuels, diesel

0001894

-6-

1 : fuels and heating oils. Low temperature flow properties
are most usually encountered with diesel fuels and
with heating oils.

5 While fuel treatment rates in excess of 0.25 wt.% may
be used, such as up to about 0.5 wt.%, excellent
results are usually achieved within the aforesaid
range of 0.005 to 0.25 wt.% and preferred in the
range of about 0.005 to 0.05 wt.% based upon the
weight of distillate fuel.

10 The nitrogen containing wax crystal growth inhibitors
used in the present invention are generally those
having a total of 30-300, preferably 50-150 carbon
atoms and being those oil-soluble amine salts and
amides formed by reacting at least 1 molar portion of
15 a hydrocarbyl substituted amine with 1 molar portion
of the aromatic or cycloaliphatic polycarboxylic
acid, e.g. 2 to 4 carboxyl groups preferably dicarboxylic
acids, or their anhydrides or partial esters of
dicarboxylic e.g. mono-esters of dicarboxylic acids.

20 The amines may be primary, secondary, tertiary or
quaternary, but preferably are secondary. Tertiary
and quaternary amines can only form amine salts.
Examples of amines include tetradecyl amine, cocoamine,
hydrogenated tallow amine and the like. Examples of
25 secondary amines include cocomethyl amine, dioctadecyl

0061894

-7-

1 amine, methyl-benhenyl amine and the like. Amine
mixtures are also suitable and many amines derived
from natural materials are mixtures. The preferred
amine is a secondary hydrogenated tallow amine of
5 the formula HNR_1R_2 wherein R_1 and R_2 are
alkyl groups derived from tallow fat composed of
approximately 4% C_{14} , 31% C_{16} , 59% C_{18} .

Examples of suitable carboxylic acids (and their
anhydrides) include cyclohexane dicarboxylic acid,
10 cyclohexene dicarboxylic acid, cyclopentane dicarboxylic
acid, naphthalene dicarboxylic acid, and the like.
Generally these acids will have about 5-13 carbon
atoms in the cyclic moiety. Preferred acids useful
in the present invention are benzene dicarboxylic
15 acids such as phthalic acid, terephthalic acid, and
isophthalic acid. Isophthalic acid or its anhydride
is the particularly preferred embodiment.

It is preferred that the nitrogen containing compound
has at least one straight chain alkyl segment extending
20 from the compound containing 8-40 preferably 14-24
carbon atoms. Preferably the nitrogen compound
contains at least three alkyl chains each containing
from 8 to 40 carbon atoms and preferably at least two
of these chains are normal. Also at least one ammonium

-8-

1 salt, amine salt or amide linkage is required to be
present in the molecule. The particularly preferred
amine compound is the amide-amine salt formed by
reacting 1 molar portion of phthalic anhydride with 2
5 molar portions of di-hydrogentated tallow amine.
Another preferred embodiment is the diamide formed by
dehydrating this amide-amine salt.

Also suitable are the amide or amine salts of monoesters
of the aforesaid dicarboxylic acids, the alkyl chain
10 of the ester containing about 8 to 40 carbon atoms.
But lower alkyl monoesters may also be suitable
provided the nitrogen compound is an oil-soluble
compound and has about 30-300 preferably 50-150
carbon atoms. An octadecyl ester of an amine salt of
15 phthalic anhydride is an example of a preferred
embodiment in this category.

In this invention both the type of nitrogen-containing
compounds and the type of ethylene vinyl acetate
copolymer used have been found to be significant
20 parameters to provide an effective two-component
additive system which is a superior flow improver.
Thus, for example, it has been found that the flow
improver combination of the present invention is a
highly effective one compared with three-component
25 systems such as disclosed in U.S. Patent 4,211,534

-9-

0061894

1 which are used at relatively high treatment concentrations. It has been found in the present invention that the use of a third component with its associated costs may not be necessary in many fuels.

5 It is believed that the nitrogen containing compounds of the present invention are highly effective in inhibiting the growth of wax crystals. Typically as a distillate fuel cools normal alkanes containing from about 14 to 32 carbon atoms crystallise out, the
10 longer alkanes crystallising first, generally the maximum is at around 20 to 22 carbon atoms. The nitrogen containing compounds appear to be highly effective in controlling the growth of the bulk of the alkane waxes but appear to be slightly less
15 effective in controlling the initial stages of wax precipitation.

Although the optimum polymer properties will vary from one fuel to another, we prefer that the ethylene vinyl acetate copolymer contain from 10 to 40 wt.%
20 more preferably 10 to 35 wt.%, most preferably from 10 to 20 wt.% vinyl acetate; has a number average

0001037

-10-

1 molecular weight (M_n) as measured by Vapour Phase
Osmometry within the range of about 1,000 to 30,000,
preferably 1500 to 7000 more preferably 1500 to 5500
most preferably of 2500 to 5500 and a degree of
5 branching in the range of 1 to 20 preferably 2 to 12.
The degree of branching is the number of methyl
groups other than those of the vinyl acetate in the
polymer molecule per 100 methylene groups as determined
by proton nuclear magnetic resonance spectroscopy as
10 for example using a Perkin-Elmer R-34 Spectrometer on
20% (W/W) solution in orthodichlorobenzene at 100°C
operating at 220 MHz in the continuous wave mode.

Whilst the polymer branching may vary within these
limits we have found that the more important character-
15 istic of the copolymer is the vinyl acetate content.
We have found that the use of ethylene vinyl acetate
co-polymers of different solubility characteristics
due to a polymer structure especially a vinyl acetate
content outside that described above can result in a
20 fuel having adverse flow and filterability performance.

-11-

1 We have also found that the relative proportions of
the nitrogen containing compound and the ethylene
vinyl acetate copolymer is important in achieving the
improvement in flow and filterability. We have found
5 that, based on the total weight of additive in the
fuel, at least 25 wt.% preferably at least 50 wt.% of
the nitrogen containing compound should be used and
more preferably between 25 and 95 wt.% preferably 50
to 95 wt.% most preferably between 60 and 90 wt.%,
10 especially between 60 and 80 wt.% the balance
being the ethylene/vinyl acetate copolymer.

The additive systems of the present invention may
conveniently be supplied as concentrates of the
mixture of the nitrogen containing compound and the
15 ethylene vinyl acetate copolymer in oil or other
suitable inert solvent for incorporation into the
bulk distillate fuel. These concentrates may also
contain other additives as required. These concentrates
which contain from 3 to 90 wt.% preferably from 3 to
20 60 wt.%, more preferably 10 to 50 wt.% of the

0061894

-12-

1 additives in oil or other solvent are also within the
scope of the present invention.

The invention is further illustrated by the following
examples which are not to be considered as limitative
5 of its scope. In these Examples unless specified
otherwise reference to parts is parts by weight.

In the Examples 1 to 11 below the fuel has been
evaluated according to the Distillate Operability Test
(DOT test) which is a slow cooling test shown to be
10 reasonably accurate compared with actual field conditions.

DOT Test

Flow Improved Distillate Operability Test (DOT test)
is a slow cooling test designed to correlate
with the pumping of a stored heating oil. The cold
15 flow properties of the described fuels containing the
additives were determined by the slow cool flow test
as follows. 300 ml of fuel are cooled linearly at 1°C/hour
to the test temperature then that temperature is held
constant. After 2 hours at the test temperature, approx-
20 imately 20 ml of the surface layer is removed by suction
to prevent the test being influenced by the abnormally
large wax crystals which tend to form on the oil/air
interface during cooling. Wax which has settled

0061894

-13-

1 in the bottle is dispersed by gentle stirring, then a CFPP
filter assembly as described hereafter in relation to CFPP
Test is inserted. A vacuum of 300 mm of water is applied
and 200 ml of the fuel is passed through the filter into the
5 graduated receiver, A PASS is recorded if the 200 ml are
collected within sixty seconds through a given mesh size or
a FAIL if the filter becomes blocked and the flow rate is
too slow.

Filter assemblies with filter screens of 20, 30, 40, 60,
10 80, 100, 120, 150, 200, 250, 350 mesh number are used to
determine the finest mesh number that a wax containing fuel
will pass. The smaller are the wax crystals and therefore
the finer the mesh the greater the effectiveness of the
additive flow improver. It should be noted that no two
15 fuels will give exactly the same test results at the same
treatment level for the same flow improver additive, and,
therefore, actual treat levels will vary somewhat from fuel
to fuel.

"Nitrogen Compound A"

20 The amide/dialkyl ammonium salt from the reaction product
of 1 mole of phthalic anhydride with 2 moles of a
secondary di(hydrogenated tallow) amine, containing a
mixture of tallow fat n-alkyl groups as follows: 4%
C₁₄, 31% C₁₆, and 59% C₁₈.

0061894

-14-

1 "EVA Polymer 1"

Was an ethylene-vinyl acetate copolymer of \overline{M}_n 3400

"V.P.O.", having 17.0 wt.% vinyl acetate and an 8.0 degree of branching i.e. 8 methyl terminating alkyl

5 side chains other than vinyl acetate per 100 methylene groups.

The characteristics of the fuels used in the following Examples were

<u>Fuel</u>	<u>Distillation (ASTM D86), °C</u>				<u>Cloud</u>	<u>Wax</u>
	<u>IBP</u>	<u>20%</u>	<u>90%</u>	<u>FBP</u>	<u>Point</u>	<u>Appearance</u>
					<u>(°C)</u>	<u>Point</u>
						<u>(°C)</u>
1	182	220	354	385	+1	-2.5
2	180	226	341	368	-3.5	-5.5
3	188	238	344	375	-1	-4.5

0061894

-15-

1 Example 1

Fuel 1 was evaluated in the DOT test using a flow improver composed of 75% by weight of Nitrogen Compound A, and 25% by weight of EVA Polymer 1 and the results at -12°C are reported below:

5	<u>Concentration in Fuel</u>	<u>Finest Mesh Passed</u>
	100 ppm	80
	150 ppm	350
	200 ppm	350

Example 2

10 Example 1 was repeated but using Fuel 2 with the following results:

	<u>Concentration in Fuel</u>	<u>Finest Mesh Passed</u>
	50 ppm	40
	150 ppm	200
15	200 ppm	250

Example 3 - Comparison

For the purpose of comparison, the test of Example 1 was conducted with the conventional flow improver additive as reported in Example 1, polymer 1 in U.S. Patent 4,211,534. This flow improver additive is described as a polymer mixture of about 75 wt.% of a wax growth arrestor and about 25 wt.% of a nucleator, both compounds being ethylene vinyl acetate polymer, henceforth referred to as Polymer 15.

20

0061894

-16-

<u>ppm of Additive</u>	<u>Finest Mesh Passed</u>	
	Fuel 1	Fuel 2
100	40	30
150	100	40
200	120	80

1 Example 4

(a) The test of Example 2 was repeated in Fuel 2 using a flow improver composed of 100 parts by weight

Nitrogen Compound A and 25 parts by weight of EVA

5 Polymer 1. 125 ppm of this was added to the fuel and the finest filter mesh passed was 200.

(b) Example 4(a) was repeated except that 25 parts of an ethylene vinyl acetate copolymer having a M_n of

2000 and a 36% vinyl acetate content was added to the

10 composition of Example 4(a) to thereby provide a three component additive and the finest filter mesh passed was 120. This indicates the adverse results of adding components heretofore considered desirable to the two component system of this invention.

15 Example 5

The DOT test used in Example 1 was repeated using Fuel 3.

All tests were at -12°C with 100 ppm flow improver composed of 75 ppm Nitrogen Compound A of Example 1 and 25 ppm of various ethylene vinyl acetate copolymers (EVA)

20 tabulated below. Results are in Table 1. The purpose of this example is to demonstrate the importance of the particular category of ethylene-vinyl acetate copolymers.

0061894

-17-

TABLE 1

<u>Polymer</u>	<u>Wt. % VA</u>	<u>M_n</u>	<u>Branching*</u>	<u>Finest Mesh Passed</u>
2.	13.5	2750	9.1	80
3	15.8	5500	7.6	100
4	17.0	3400	8.0	150
5	27.6	6250	5.6	100
6	29.4	3050	9.1	60
7	33.0	5000	10.0	60
8	36.0	2000	4.0	60

- 1 * Branching is the number of methyls per 100 methylene
groups excluding the vinyl acetate methyls as measured
by ^1H NMR Nuclear Magnetic Resonance spectroscopy.
All spectra were run on a Perkin-Elmer R-34 spectrometer
5 on 20%(w/w) solution in orthodichlorobenzene at 100°C
operating at 220 MHz.

Example 6

- The performance of an additive mixture containing 3 parts by weight of Nitrogen compound A and 1 part by weight of EVA Polymer 1 was compared at different concentrations of additive with

- | | | | |
|-------|--------------------------|---|---|
| (i) | Polymer 15 | - | B |
| (ii) | EVA Polymer 7 on its own | - | C |
| (iii) | EVA Polymer 8 of Table 1 | - | D |

0061894

-18-

1 The results in the DOT test at -12°C in Fuel 1
are shown in Figure 1 those for the composition of the
invention being curve A, the lettering of the other curves
correspond to the above Table.

5 Examples 7 and 8

The comparison of Example 6 was repeated in Fuels 2 and 3
and the results are shown in Figures 2 and 3 respectively.

Example 9

Mixtures of different proportions of Nitrogen compound A and
10 EVA Polymer 1 were prepared and tested in Fuel 1 in the DOT
test at -12°C and treat rates of 200 and 125 ppm
additive in the fuel. The results were compared with a
similar additive mixture but containing EVA Polymer 8
of Table 1. The results are shown in Figure 4, the
15 upper curve being at 200 ppm additive treat rate, the
lower at 125 ppm. In each curve trace E is of the
present invention and trace F is the Composition
containing EVA Polymer 8 of Table 1 in the place of
EVA Polymer 1.

20 Examples 10 and 11

Example 9 was repeated but using Fuels 2 and 3 and the
results are shown in Figures 5 and 6 respectively.

-19-

0061894

1 In the following Examples 12 to 16 the response of the
oil to the additives was measured by the Cold Filter
Plugging Point Test (CFPPT) which is carried out by
the procedure described in detail in "Journal of the
5 Institute of Petroleum", Volume 52, Number 510, June-
1966, pp. 173-185. This test was designed to correlate
with the cold flow of a middle distillate in European
automatic diesels.

In brief, a 40 ml sample of the oil to be tested is
10 cooled in a bath which is maintained at about -34°C to
give non-linear cooling at about $1^{\circ}\text{C}/\text{min}$. Periodically (at
each one degree Centigrade drop in temperature starting
from at least 2°C above the cloud point) the cooled
oil is tested for its ability to flow through a fine
15 screen in a prescribed time period using a test device
which is a pipette to whose lower end is attached an
inverted funnel which is positioned below the surface
of the oil to be tested. Stretched across the mouth
of the funnel is a 350 mesh screen having an area
20 defined by a 12 millimetre diameter. The periodic
tests are each initiated by applying a vacuum to the
upper end of the pipette whereby oil is drawn through
the screen up into the pipette to a mark indicating 20
ml of oil. After each successful passage the oil is
25 returned immediately to the CFPP tube. The test is
repeated with each one degree drop in temperature
until the oil fails to fill the pipette within 60

0061894

-20-

1 seconds. This temperature is reported as the CFPP temper-
ature. The difference between the CFPP of an additive
free fuel and of the same fuel containing additive is
reported as the CFPP depression by the additive. A more
5 effective additive flow improver gives a greater CFPP
depression at the same concentration of additive.

Example 12

The CFPP performance of Fuel 1 containing various con-
centrations of the following additives was measured and
10 recorded on the curves of Figure 7.

	Additive	Curve
	(i) Nitrogen Compound A	G
	(ii) EVA Polymer 8 of Table 1	H
	(iii) EVA Polymer 1	I
15	(iv) Polymer 15	J
	(v) 3 Parts Nitrogen Compound A 1 Part EVA Polymer 1	K

Example 13 and 14

The evaluations of Example 12 were repeated in Fuels 2 and
20 3 and the results are recorded in Figures 8 and 9
respectively.

-21-

0061894

1 Example 15

The CFPP performance of Fuel 1 containing 50 ppm and 100 ppm of mixtures of different proportions of Nitrogen Compound A and EVA Polymer 1 were determined and recorded on the attached Figure 10.

Examples 16

Example 15 was repeated but using Fuels 2 and 3 and the results are recorded in Figures 11 and 12 respectively.

Example 17

10 The additive combinations of the present invention were evaluated in Fuels 4 and 5 which had the following characteristics

	<u>Fuel 4</u>	<u>Fuel 5</u>
ASTM Cloud Point, °C	-15	-10
Pour Point, °C	-21	-24
WAP, °C	-17.5	-15
Distillation, °C		
Initial Boiling Point	179	158
10%	215	203
20%	230	225
50%	263	269
90%	314	320
Final Boiling Point	345 (98.2%)	347
Residue %	1	1.1

0061894

-22-

- 1 The performance of the additives is evaluated in a test developed for the low temperature properties of diesel fuels in which a sample of the fuel is brought to the test temperature by cooling at 2°F per hour and testing
- 5 the filterability at that temperature by determining if the fuel will pass through a 350 mesh screen under a vacuum of 6 inches of mercury within 60 seconds. If so the fuel is considered to PASS.

The ethylene vinyl acetate copolymers used in this Example

10 had the following structure

TABLE 2

<u>Polymer</u>	<u>MW (VPO)</u>	<u>% VA</u>	<u>Methyl Branching</u>
9	5600	36.2	8.5
10	5000	17	7.5
11	3050	29.4	9.1
12	2775	17.1	8.2
13	2000	36	4
14	1950	29.1	4.6

0061894

-23-

- 1 Mixtures of Nitrogen Compound A with varying amounts of ethylene vinyl acetate copolymers 9 to 14 were tested in Fuels 4 and 5, the amount of additive needed to PASS the test being recorded in Figures 13 and 14 respectively.
- 5 The lower the amount of additive showing the better performance of the additive.

The numbers on the curves refer to the number given to the ethylene vinyl acetate copolymer in Table 2 above.

- A Fuel 7 having the following characteristics was used in
- 10 the next 2 Examples.

	Cloud Point (°C)	-2
	Wax Appearance Point (°C)	-6
	Distillation (ASTM D-86) (°C)	
	IBP	164
15	20	212
	50	262
	90	333
	FBP	370
	Aromatics (% (v/v))	28

20 Example 18

Two three cubic metre tanks of the Fuel 7 were cooled under ambient conditions to -14°C and after a cold soak period a 300 ml sample of the fuel was tested for its cold flow performance, as in the DOT. The barrels were

25 then slowly heated to above the WAP of the fuel then

0061894

-24-

1 cooled again at 0.5°C/hour to -14°C. The fuel was then pumped out of the barrels through a range of filter screens to determine the finest that the waxy fuel could pass through.

5 The fuel in one barrel contained 135 parts per million of Polymer 15 and only passed a 30 mesh screen whilst the fuel in the other barrel which contained 135 parts per million of a mixture of 4 parts of Nitrogen Compound A and 1 part of EVA Polymer 1 passed a 100 mesh screen.

10 Example 19

In this example, the results are from four 25 m³ tanks of Fuel 7 which were tested side by side. Over a period of three weeks storage, under natural cold conditions (including natural temperature cycling), the fuel at -14°C
15 was pumped out of the tanks as in a fuel distribution situation - and the finest filter screen that the fuel would flow through was recorded as follows

	Treat Rate P.P.M.	Additive	Mesh Passed
20	70	Polymer 15	30
	70	4 Parts Nitrogen Compound A 1 Part EVA Polymer 1	40
	135	Polymer 15	30
	135	4 Parts Nitrogen Compound A 1 Part EVA Polymer 1	100

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